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(57) Abstract

The subject invention provides water-swellable polymer compositions having reduced caking tendencies. Such polymer compositions can be produced by employing quaternary ammonium salts. In addition to having reduced caking tendencies such polymers have reduced dust and substantially maintain or increase the surface tension of an aqueous fluid in equilibrium with the polymer and air.

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ABSORBENT POLYMERS HAVING A REDUCED CAKING TENDENCY

This invention relates to absorbent polymers whose particles have a reduced tendency to agglomerate in humid environments and/or form dust, a process for the preparation of such polymers, and absorbent articles prepared therefrom.

Absorbent, that is, water-swellable, polymers are generally prepared by gel polymerization of a mixture of monomers in aqueous solution. Certain additives, such as crosslinking agents, may be incorporated into the monomer mixture. The product of the polymerization process is then typically dried and subjected to a mechanical means of particle size reduction.

The absorbent polymers are especially useful in many types of personal care devices such as diapers, adult incontinent articles, sanitary napkins, and medical devices because of their ability to readily absorb bodily fluids. Unfortunately, the absorbent polymers also absorb water from the air and when exposed to humid environments the water swellable polymer particles tend to stick together, that is, agglomerate or cake, and also tend to stick or adhere to processing equipment. Thus, the polymer particles are not free-flowing, that is, have poor flowability.

[illegible] the polymer particles are not free-flowing [illegible] present a few problems. One problem is that the [illegible] difficult to incorporate into personal care [illegible] use the reduced flowability hinders the [illegible] distribution of particles within an absorbent core. Another problem stems from the tendency of the particles to stick to each other and to the manufacturing and processing equipment, that is, screens, dryers, meters, or fabricating machinery. Production is often hindered because the equipment must be cleaned periodically to remove the agglomerated particles. Yet another problem associated with water-swellable polymer

particles that tend to agglomerate is that if the polymer particles have absorbed some water already then the water-swelling capacity for the personal care devices containing said polymer particles may be reduced.

One way in which the caking tendencies of absorbent polymers have been reduced is by blending finely divided silica or fumed silica with the absorbent polymer particles. See, for example, U.S. Patent 4,734,478 which mixes 0.01 to 10 parts by weight of finely divided silica with 100 parts by weight of the water absorbent polymer and WO 94/22940 which blends less than 10 percent by weight fumed silica with the water absorbent polymer. Unfortunately, even though the caking tendency of the polymer particles may be reduced, both types of silica are low bulk density solids and tend to add dust into the environment.

Other compounds have been employed to reduce the adhesion tendency of absorbent polymer particles. Among these are nonionic or anionic surfactants. See U.S. Patent 4,286,082 utilizes the above surfactants in the presence of a crosslinkable monomer to diminish the adhesion of the hydrous polymer to the polymerization vessel. These surfactants may indeed reduce the adhesion tendency of the polymer but the surfactants also reduce the surface tension of a contacting fluid in equilibrium with the polymer. This is unfortunate in that the surface [illegible]he contacting fluid is used as a measure of [illegible]f water-swellable polymers.

[illegible] surface tension of the contacting fluid in [illegible] is used as a measure of performance because water-swellable polymers which exhibit a higher surface tension generally tend to "wick" better, that is, the polymer transports aqueous fluids by capillary flow more efficiently and holds the fluids more strongly. As disclosed in U.S. Patent 5,352,711, wicking is very important for absorbent devices such as diapers or sanitary articles. The effect of surface tension on

capillary flow and capillary pressure is described more fully by the Lucas-Washburn equation as discussed in P.K. Chatterjee, "Absorbency," Elsevier, Amsterdam, 1985, pp. 36-37.

Industry would find great advantage in a water-swellable polymer composition which does not agglomerate when exposed to a humid environment, that is, which comprises more than 90 percent free-flowing polymer particles as measured by the test disclosed herein. Industry would further find advantage in a new process for reducing the agglomeration tendencies of a water absorbent polymer composition which comprises employing liquid anticaking agents rather than solids such as silica which can increase the dust present. Industry would further find advantage if said liquid anticaking agents substantially maintain or increase the surface tension of a contacting liquid in equilibrium with the polymer composition.

Accordingly, in one aspect the subject invention provides a composition comprising: a) water-absorbent lightly crosslinked water-insoluble polymer particles; b) an effective amount of an anticaking agent; and optionally c) a hydrophobic or hydrophilic dedusting agent. These compositions comprise more than 75 percent, preferably more than [illegible] percent, more preferably more than 95 percent, and most preferably more than 99 percent non-[illegible]mer particles as measured by the test disclosed [illegible]

[illegible] another aspect, the invention also provides a [illegible] comprising: a) preparing a water-swellable hydrogel by a gel polymerization process; b) drying and sizing the thus prepared hydrogel to obtain a composition comprising dried and sized particles; and c) contacting the composition with an effective amount of an anticaking agent.

Quaternary ammonium salts have been used in conjunction with absorbent gels as a lubricant in, for example, U.S. Patent 4,997,714, but heretofore have not been employed as anti-caking agents.

As used herein, the term "substantially maintain or increase the surface tension" means that when an aqueous fluid is in equilibrium with another fluid and the attrition-resistant, water-swellable polymers treated with a hydrophobic dedusting agent of the present invention, the surface tension of the aqueous fluid, as measured by a surface tension test disclosed hereinafter, is decreased by less than about 10 percent, preferably less than about 5 percent, when compared to water-swellable polymers that are not contacted with a hydrophobic dedusting agent. More specifically, the aqueous fluid employed in the surface tension test disclosed herein is a 0.9 percent NaCl solution and the other fluid is air.

As used herein, the term "unassociated dust" means the measurable portion of a water absorbent polymer particle composition having a maximum diameter less than or equal to 10 microns which portion is rendered airborne when air is applied to the composition. More specifically, the air is applied and the portion is measured using a pulsed-jet disperser described below.

As used herein, the term "physical coating" [illegible] coating of the anticaking agent and the [illegible]t (if present) in contact with the polymer [illegible] dust (if present), is not chemically reacted [illegible]er, for example, no covalent surface [illegible] reaction occurs between the anticaking or dedusting agent and the polymer particles or dust.

As used herein, the term "aliphatic hydrocarbon" includes straight- or branched-chain alkyl, alkenyl, and alkynyl groups which may have linkages such as silicon (Si) or oxygen (O) within the chain. The aliphatic

hydrocarbons may be unsubstituted or substituted with inert substituents.

As used herein, the term "aryl group" includes condensed aromatic rings such as phenyl, naphthyl, phenanthryl, and anthracenyl which are unsubstituted or substituted with inert substituents.

As used herein, the term "inert substituent" means a substituent which is inert, that is, non-reactive to its environment. The environment to which the substituent is inert includes the water-absorbent polymer particles and the aqueous liquid in which it is to be dissolved or dispersed. Included among inert substituents are, for example, halogen groups such as fluoro, chloro, and bromo, hydroxy groups, and alkoxy groups.

The water-swellable or lightly crosslinked hydrophilic polymers that are employable in the present invention can be any of the known hydrophilic polymers which are capable of absorbing large quantities of fluids. In particular, water-absorbent polymers useful in this invention are water-absorbent polymers which contain carboxyl moieties. Preferably, at least 0.01 equivalent of carboxyl groups were present per 100 grams of the water-absorbent resin.

Among preferred carboxyl-containing water absorbent polymers are hydrolyzates of starch-[illegible] graft copolymers, partially neutralized [illegible] starch-acrylic acid or starch-polyvinyl [illegible] copolymers, saponification products of vinyl [illegible] ester copolymers, derivatives of copolymers of isobutylene and maleic anhydride, hydrolyzates of acrylonitrile copolymers, crosslinked products of hydrolyzates of acrylonitrile copolymers, crosslinked carboxy-methyl cellulose, hydrolyzates of acrylamide copolymers, crosslinked products of hydrolyzates of acrylamide copolymers, partially

neutralized products of polyacrylic acids and crosslinked products of partially neutralized polyacrylic acids.

Examples of some suitable polymers and processes, including gel polymerization processes, for preparing them are disclosed in U.S. Patents 3,997,484; 3,926,891; 3,935,099; 4,090,013; 4,093,776; 4,340,706; 4,446,261; 4,683,274; 4,459,396; 4,708,997; 4,076,663; and 4,190,562. Such hydrophilic polymers can be prepared from water-soluble α,β-ethylenically unsaturated monomers such as monocarboxylic acids, polycarboxylic acids, acrylamide and their derivatives. Suitable α,β-ethylenically unsaturated monomers include, for example, acrylic acid, methacrylic acid, crotonic acid, isocrotonic acid and alkali metal salts and ammonium salts thereof; itaconic acid, acrylamide, methacrylamide and 2-acrylamido-2-methyl-1-propane sulfonic acid and its salts. The preferred monomers include acrylic acid and methacrylic acid and their respective salt forms such as alkali metal or ammonium salts. The water-soluble monomers useful in the present invention may be used in amounts ranging from [illegible] percent to 80 percent by weight based on the total weight of the aqueous monomer solution. Preferably, the amount ranges from [illegible] percent to 60 percent based on the total weight of the aqueous monomer solution.

Optionally, minor amounts of other water-soluble unsaturated monomers, such as alkyl esters of [redacted] for example, methyl acrylate or methyl [redacted] may be present in the water absorbent [redacted] addition, certain grafting polymers, such as, [redacted] polyvinyl alcohol, starch and water soluble or swellable cellulose ethers may be employed to prepare products having superior properties. Such grafting polymers, when employed, are used in amounts up to about 10 weight percent based on the α,β-ethylenically unsaturated monomer. Further, it may be advantageous to include a chelating agent to remove trace metals from solution, for example, when a metal reaction vessel is employed. One such chelating agent is VERSENEX™ V 80

(Trademark of The Dow Chemical Company) an aqueous solution of the pentasodium salt of diethylenetriamine pentaacetic acid. Such chelating agents, when employed, are generally used in amounts between 100 and 2000 ppm based on the α,β-ethylenically unsaturated monomer.

It is desirable to obtain a level of conversion of monomer to polymer of at least about 95 percent. The polymerization may be carried out using acid monomers that are not neutralized or that have been neutralized or partially neutralized prior to the polymerization. Neutralization is conveniently achieved by contacting the aqueous monomer with an amount of basic material sufficient to neutralize between 20 and 95 percent of the acid groups present in the acid monomers. Preferably, the amount of basic material will be sufficient to neutralize between 40 percent and 85 percent, and most preferably between 55 percent and 75 percent of the acid groups present in the acid monomers. When pre-neutralizing the monomer solution, it is important to control the neutralization conditions so that the heat of neutralization does not cause the premature polymerization of the monomer mixture. The neutralization is advantageously carried out at temperatures below 40°C, preferably at temperatures below 35°C.

Compounds which are useful to neutralize the acid groups of the monomer are typically those which will [illegible] neutralize the acid groups without having a [illegible] effect on the polymerization process. Such [illegible]nclude alkali metal hydroxides, and alkali [illegible]nates and bicarbonates. Preferably, the material used to neutralize the monomer is a sodium or potassium hydroxide or carbonate. In determining the desired degree of neutralization, care must be taken to ensure that the pH of the resulting crosslinked absorbent polymer, which will be contacted with or dispersed in an aqueous fluid to be absorbed, is maintained in a range appropriate for the applications for which the polymer is intended. Alternatively, the polymerization may be

carried out employing unneutralized monomers and thereafter neutralizing, as is known in the art.

Conveniently, a conventional vinyl addition polymerization initiator is used in the polymerization of the water-soluble monomers and the crosslinking agent. A free radical polymerization initiator which is sufficiently soluble in the monomer solution to initiate polymerization is preferred. For example, water soluble persulfates such as potassium persulfate, ammonium persulfate, sodium persulfate, and other alkali-metal persulfates, hydrogen peroxide and water soluble azo-compounds such as 2,2'-azobis (2-amidinopropane.HCl) may be used. Some of these initiators, such as hydrogen peroxide, can be combined with reducing substances such as sulfites or amines to form known redox initiator systems. The total amount of initiators used may range from 0.01 to 1.0 weight percent, preferably 0.01 to 0.5 weight percent, based on the total weight of α,β ethylenically unsaturated monomer reactants.

The water-absorbent resin will preferably be lightly crosslinked to render it water-insoluble and water-swellable. The desired crosslinked structure may be obtained by the copolymerization of the selected water-soluble monomer and a crosslinking agent possessing at least two polymerizable double bonds in the molecular unit. The crosslinking agent is present in an amount [illegible] crosslink the water-soluble polymer. The [illegible]nt of crosslinking agent is determined by [illegible]egree of absorption capacity and the desired [illegible] retain the absorbed fluid, that is, the desired absorption under load (AUL). Typically, the crosslinking agent is used in amounts ranging from 0.0005 to 5 parts by weight per 100 parts by weight of α,β-ethylenically unsaturated monomer used. More preferably, the amount ranges from 0.1 to 1 part by weight per 100 parts by weight of the α,β-ethylenically unsaturated monomer. Usually, if an amount over about 5 parts by weight of crosslinking agent per 100 parts is used, the

resulting polymer has too high a crosslinking density and exhibits a reduced absorption capacity and increased strength to retain the absorbed fluid. If the crosslinking agent is used in an amount less than about 0.0005 part by weight per 100 parts, the polymer usually has too low a crosslinking density, and when contacted with the fluid to be absorbed becomes sticky and exhibits a lower initial absorption rate.

While the crosslinking agent will typically be soluble in the aqueous solution of the α,β-ethylenically unsaturated monomer, the crosslinking agent may be merely dispersible in such a solution, without negative implications. The use of such dispersing agents is disclosed in U.S. Patent 4,833,222. Suitable dispersing agents include carboxymethyl cellulose suspending aids, methyl cellulose, hydroxypropyl cellulose, and polyvinyl alcohol. Such dispersing agents are typically provided at a concentration between 0.005 and 0.1 weight percent, based on the total weight of α,β-ethylenically unsaturated monomer reactants.

Typical crosslinking agents include monomers having in one molecule 2 to 4 groups selected from CH_2=CHCO-, CH_2=C(CH_3)CO- and CH_2=CH-CH_2-. Exemplary crosslinking agents are diacrylates and dimethacrylates of ethylene glycol, diethylene glycol, triethylene glycol, propylene glycol, 1,4-butanediol, 1,5-pentanediol, 1,6-[illegible] neopentyl glycol, trimethylolpropane and [illegible], triacrylates and trimethacrylates of [illegible]propane and pentaerythritol; highly ethoxylated [illegible]propane triacrylate; tetraacrylate and tetramethacrylate of pentaerythritol; and tetraallyloxyethane. As noted in WO 93/05080, published on March 18, 1993, certain crosslinking agents yield particularly preferred absorptive properties. Such preferred crosslinking agents include methylenebisacrylamide, bis(acrylamido)acetic acid and its salts, allyl acrylate, allyl methacrylate, and esters or amides having both a vinyl and an allyl functionality.

Other particularly preferred crosslinking agents and methods include those disclosed in WO 94/20547, published on September 15, 1994. Such preferred crosslinking agents include mixtures of polyvinyl compounds such as, for example, highly ethoxylated trimethylolpropane triacrylate and allyl methacylate, and polyglycols such as, for example, polyethylene glycol.

In a preferred embodiment for making polymers useful in the practice of this invention, an aqueous solution of the α,β-ethylenically unsaturated monomer in the partially neutralized form, the crosslinking agent, the initiator and a grafting polymer substrate, if desired, is prepared. The polymerization of the mixture may be carried out by methods well known to those skilled in the art. The resultant polymer is typically pre-sized and dried using means well known in the art.

To improve absorptive properties, the dried particles may be heat treated in accordance with the procedures set forth in WO 93/05080, published on March 18, 1993, and/or WO 94/20547, published on September 15, 1994. In particular, the dried particles are heated for a time sufficient to increase the modulus, centrifuge capacity, and/or the absorbency under load. An oxidizing agent, such as a bromate, chlorate, chlorite, or mixture thereof, may be uniformly distributed within the water absorbent polymer prior to such heat treatment to enhance [redacted] the preceding properties. Such heat [redacted] preferably carried out at a temperature of at [redacted] more preferably of at least 180°C, and most [redacted] at least 190°C. Such heat treatment is preferably carried out at a temperature of less than 250°C, more preferably less than 240°C.

The time period for heat treatment should be sufficient to effect an improvement in absorptive properties. The exact times of heat treatment required will be affected by the equipment chosen, and can be determined empirically by examination of product

properties. Preferably, the time is at least 3 minutes, and more preferably at least 5 minutes. If the time is too long, the process becomes uneconomical and a risk is run that the absorbent resin may be damaged. Preferably, the maximum time of heating is 150 minutes or less, more preferably 60 minutes or less.

The method of heat treatment is not critical. For example, forced air ovens, fluidized bed heaters and heated screw conveyors may be successfully employed. If desired, the heated polymer may be remoisturized for ease in handling. While such remoisturization may serve to decrease the amount of unassociated dust, it may lead to clumping of the polymer product.

Another way to improve absorptive properties of the polymer particles may be to surface crosslink the polymer particles. Procedures for surface crosslinking are well known in the art and described in, for example, DE 4244548, DE 4020780, EP 605150, and U.S. Patents 4,734,478 and 4,666,983. These procedures, like heat treatment, may increase the modulus, centrifuge capacity, and/or the absorbency under load of the polymer particles.

The dried, sized and optionally heat-treated or optionally surface crosslinked polymer particles are then contacted with an effective amount of an anticaking agent. It is critical that the polymer particles not be in the [illegible] gel, that is, have less than 30, preferably less [illegible] re preferably less than about 10 weight percent [illegible] re contacting them with the anticaking agent. [illegible] caking agent is contacted with polymer particles in gel form then the anticaking agent is likely to combine with water and be absorbed further into the interior of the polymer particles. If this occurs then the anticaking properties of the particles are reduced because the anticaking agent is not at the surface of the particles.

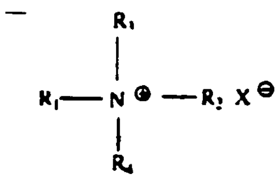
It is also critical to this invention that the polymer particles not be subjected to temperatures above 100°C after the particles are contacted with the anticaking agent. This is due to the fact that the anticaking agent may vaporize or degrade at higher temperatures. Therefore, if a drying step and/or a heat treatment step is to be undertaken which will subject the particles to temperatures above 100°C, then the drying and/or the heat treatment steps should be accomplished before the particles are contacted with the anticaking agent.

The anticaking agent physically coats the polymer particles and serves to diminish the adhesion of the polymer particles to each other and to machinery or vessels with which the polymer particles come into contact. The polymer particles will not suffer significant decreases in flowability nor undergo significant caking upon moisture absorption. After contact with an effective amount of anticaking agent, at least 75, preferably at least 90, more preferably at least 95, most preferably at least 99 percent of the particles should be non-caking polymer particles according to the test disclosed herein entitled "Caking Test."

As defined herein, the term "effective amount of an anticaking agent" means an amount of a cationic surfactant, for example, a sulfonium, phosphonium, or [illegible]monium compound, which, when applied to the [illegible]cles, renders at least 75, preferably at [illegible] preferably at least 95, most preferably at [illegible]ent of the particles non-caking according to the "Caking Test." Although this effective amount will vary depending upon the polymer, the anticaking agent, and the amount of moisture, typically the amount is at least 100, preferably at least 500, more preferably at least 1000 parts per million of anticaking agent based on the weight of the polymer particles. Typically, this amount is at most [illegible], preferably at most 4000, more preferably

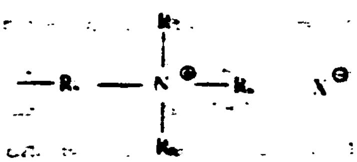
at most 3000 parts per million of anticaking agent based on the weight of the polymer particles.

Preferably, the cationic surfactants useful as anticaking agents are quaternary ammonium salts which are represented by the formula I:



(I)

wherein R_1 and R are independently a C_1-C aliphatic hydrocarbon, a C-C aliphatic hydrocarbon substituted with an unsubstituted aryl group, a C-C_4 aliphatic hydrocarbon substituted with an aryl group substituted with a C-C aliphatic hydrocarbon, or a phenyl group; R is a C_1-C aliphatic hydrocarbon or a phenyl group; and R_4 is a C-C aliphatic hydrocarbon, a C-C_4 aliphatic hydrocarbon substituted with an unsubstituted aryl, or a C_1-C aliphatic hydrocarbon substituted with an aryl group substituted with a C-C aliphatic hydrocarbon, or R_4 is a radical represented by the formula II:



(II)

[illegible] s a C-C alkylene group; R is a C-C_4 aliphatic [illegible] or a phenyl group; R and R are independently [illegible] hatic hydrocarbon, a C-C_4 aliphatic [illegible] substituted with an unsubstituted aryl group, a C-C_4 aliphatic hydrocarbon substituted with an aryl group substituted with a C-C aliphatic hydrocarbon, or a phenyl group; or R and R taken together with the nitrogen atom forms a ring selected from the group consisting of pyrrolidine, pyrroline, pyrrole, pyrazole, triazole, indole, piperazine, piperidine, morpholine, and hexahydroazepine, or R, R, and R_4 taken together form a ring selected from the group consisting of pyridinium and

together form a ring selected from the group consisting of pyridinium and imidazolinium; and $X^{\ominus}$ is an anion; with the proviso that if R_3 is not the radical represented by formula II, then the sum of the carbons in R_1, R_2, R_3, and R_4 must be at least 15 and if R_3 is the radical represented by formula II, then at least one of R_1, R_2, R_3 and R_4 must have 8 or more carbon atoms.

A preferred embodiment is the above formula in which R_1 is an aliphatic hydrocarbon having at least 16 carbon atoms such as hexadecyl, octadecenyl, octadecyl, docosanyl, docosenyl, and natural product derivatives such as tallow, soy, and rape seed; R_2 is an alkyl group having 1 to 6 carbons such as methyl, ethyl, 2-hydroxyethyl, 3-hydroxypropyl, 2-(2-hydroxy-ethyloxy)ethyl, or 3-(3-hydroxypropyloxy)-propyl, and R_3 and R_4 are independently an alkyl group having 1 to 6 carbons such as methyl, ethyl, 2 hydroxyethyl, 3-hydroxypropyl, 2-(2-hydroxyethyloxy)ethyl, or 3-(3-hydroxypropyloxy)-propyl or an alkyl group having 1 to 4 carbon atoms substituted with a phenyl such a benzyl or phenethyl group.

Another preferred embodiment is the above formula in which R_1 and R_2 are independently an aliphatic hydrocarbon having at least eight carbon atoms such as octyl, decyl, dodecyl, tetradecyl, hexadecyl, octadecenyl, octadecyl, docosanyl, docosenyl or natural product derivatives such as tallow, soy, and rape seed; R_3 is an [illegible] having one to six carbon atoms such as methyl [illegible]oxyethyl, 3-hydroxypropyl, 2-(2-[illegible]oxy)ethyl, or 3-(3-hydroxypropyloxy)-propyl; [illegible]lkyl group having 1 to 6 carbons such as methyl, ethyl, 2-hydroxyethyl, 3-hydroxypropyl, 2-(2-hydroxyethyloxy)ethyl, or 3-(3-hydroxypropyloxy)-propyl or an alkyl group having 1 to 4 carbon atoms substituted with a phenyl such as benzyl or phenethyl group.

Yet another preferred embodiment is the above formula wherein R_3 is the radical represented by formula II, R_1 is an aliphatic hydrocarbon having at least 8

carbon atoms such as octyl, decyl, dodecyl, tetradecyl, hexadecyl, octadecenyl, octadecyl, docosanyl, docosenyl, or natural product derivatives such as tallow, soy, and rape seed; R_2 and R_3 are independently an alkyl group having one to six carbon atoms such as methyl, ethyl, 2-hydroxyethyl, 3-hydroxypropyl, 2-(2-hydroxy-ethyloxy)ethyl, or 3-(3-hydroxypropyloxy)-propyl; R_4 is a C_1-C_6 alkylene such as ethylene, propylene, or butylene; and R_5, R_6 and R_7 are independently a C_1-C_{22} aliphatic hydrocarbon such as methyl, ethyl, 2-hydroxyethyl, 3-hydroxypropyl, 2-(2-hydroxy-ethyloxy)ethyl, or 3-(3-hydroxypropyloxy)-propyl, octyl, decyl, dodecyl, tetradecyl, hexadecyl, octadecenyl, octadecyl, docosanyl, docosenyl, or natural product derivatives such as tallow, soy, and rape seed or alkyl groups having 1 to 4 carbon atoms substituted with a phenyl such as benzyl or phenethyl.

Preferably, X^- is a halide such as chloride (Cl^-) or a methyl sulfate group ($MeSO_4^-$).

For ease of contacting the anticaking agent with the polymer particles, it is preferable to utilize the anticaking agent in a liquid form. Because the quaternary ammonium salts used as anticaking agents have relatively low melting points, the anticaking agent may simply be heated above its melting point to form a liquid which is then applied to the polymer. However, if heat is employed [illegible]perature should not be raised above the [illegible]gent's boiling point. For this reason, it may [illegible]erable to dissolve the anticaking agent in a [illegible]h is a liquid at room temperature. Although not required, it may be advantageous if the solvent which is employed is readily removed from the polymer particles, for example, by evaporation. In this manner solvents which may affect the surface tension of an aqueous fluid in equilibrium with the polymer, can be removed and the surface tension can be substantially maintained or increased. Useful solvents include such compounds as water, alcohols, ethers, ketones, and mixtures thereof.

A particularly preferred embodiment may be to utilize the hydrophilic dedusting compounds of WO 94/22940 as solvents. By employing hydrophilic compounds of WO 94/22940, such as polyether polyols, as solvents, one may obtain an anticaking compound which also has reduced dust. The solvent should be employed in an effective amount to serve as a dedusting agent as defined below. The hydrophilic dedusting compound may be employed in amounts which do not substantially affect the surface tension of an aqueous fluid in equilibrium with the polymer and air or in greater amounts if the hydrophilic dedusting compound readily evaporates.

Another particularly preferred embodiment may be to utilize hydrophobic compounds as solvents or as co-solvents with the hydrophilic compounds. Hydrophobic compounds disclosed below serve as dedusting agents while substantially maintaining or increasing the surface tension of an aqueous fluid in equilibrium with the polymer and air. Exemplary hydrophobic compounds often include aliphatic hydrocarbon oils, such as mineral oil, and alkanes or alkenes having between [illegible] and 18 carbon atoms optionally substituted with OH, -CO H or esters thereof. Natural oils such as castor, corn, cottonseed, olive, rapeseed, soybean, sunflower, other vegetable and animal oils, as well as esters, alcohols, and acids of these oils are useful in the invention as well as silicone oils [illegible] viscosities. When hydrophobic compounds are [illegible] solvents they should be employed in an [illegible]nt to serve as a dedusting agent as defined [illegible]

If a solvent for the anticaking material is employed which does not also serve as a dedusting agent, or if no solvent is employed, then it may be desirable to contact the dried and optionally heat treated or optionally surface crosslinked particles with an effective amount of a dedusting agent. The contacting with the dedusting agent may be employed either before, after, or simultaneously as the anticaking agent. The dedusting

agent will serve to adhere the dust to the larger polymer particles or to the walls of the mixing vessel or container in which the polymer is retained during handling, which will translate to reduced levels of unassociated dust in the finished polymer product at the various stages of handling. Moreover, the application of the dedusting agent to the polymer samples does not affect the performance or properties of the polymer.

Useful dedusting agents are hydrophilic compounds such as those disclosed in WO 94/22940, for example, polyether polyols, and hydrophobic compounds such as aliphatic hydrocarbon oils, such as mineral oil, and alkanes or alkenes having between 7 and 18 carbon atoms optionally substituted with -OH, -CO H or esters thereof. Natural oils such as castor, corn, cottonseed, olive, rapeseed, soybean, sunflower, other vegetable and animal oils, as well as esters, alcohols, and acids of the oils are useful as well as silicone oils of similar viscosities. The above compounds may be employed in purified form, in solutions, or in mixtures.

As defined herein, the term "effective amount of a dedusting agent" or "effective amount to serve as a dedusting agent" means an amount of a material which, when applied to the polymer material, reduces the concentration of unassociated dust having a diameter of less than 10 microns by at least 80, preferably at least 90, more [illegible]y at least 95, and most preferably by at least [illegible]nd or which produces dedusted compositions [illegible]ess than 7, preferably less than 5, more [illegible]ess than 2.5 ppm unassociated dust having a maximum diameter less than or equal to 10 microns. This amount will vary based upon the amount of dust initially present, the type of water-absorbent polymer, and the dedusting agent employed. Generally, the amount of dedusting agent is at least 100, preferably at least 200, more preferably at least 300 ppm based on the weight of the polymer particles. The amount is generally less than

4000, preferably less than 2000, more preferably less than 1000 ppm based on the weight of the polymer particles.

As mentioned above, if a dedusting agent is to be employed, then the dried, sized, and optionally heat treated, or optionally surface crosslinked polymer particles may be contacted with the dedusting agent either before, after or simultaneously as the contact with the anticaking agent. The dedusting agent may also be employed as a solvent for the anticaking agent. In any event, both the anticaking agent and the dedusting agent should be contacted with the polymer particles under conditions such that the particles can be easily coated with the agents. Preferably, such contacting will be conducted by spraying the agents onto the polymer or immersing the polymer in the agent followed by some form of mechanical distribution, such that adequate distribution of the agents on the water-absorbent resin particles occurs. If spraying, then it may be preferable to utilize an air atomizing nozzle and mix a fluidizing amount of water with the agents to better distribute the agent. Typically, the amount of water is less than 5, preferably less than 4, more preferably less than 3 weight percent based on the weight of the agents. The amount of water is more than [illegible], preferably more than [illegible] weight percent based on the weight of the agents. Examples of blending equipment processes include simple tumbling of a jar, or blending in a conical dryer, ribbon blender, drum [illegible] paddle blender. Moderate stirring, shaking, or [illegible] distance of conveying in a screw-conveyer can [illegible] for such adequate distribution of the agent [illegible] the particles, particularly if the particles are at an elevated temperature. Moderate grinding will also suffice, but is not necessary. The type of contacting employed may be the same or different for the anticaking agent and the dedusting agent if the polymer particles are contacted with the anticaking agent and dedusting agent at separate times.

The temperature of contacting the agents can be any temperature at which the agent does not evaporate, solidify, become too viscous, or significantly react with the carboxyl moieties of the absorbent resin polymer. Such temperatures are typically from 20°C to 150°C, preferably from 20°C to 60°C. It should be noted that elevated temperatures, that is, those above ambient temperature, that is, above 25°C, improve the speed of coating of the particles. However, if the temperature employed is between 100°C and 150°C then the time of contacting should not be so long that the agents degrade. Typically, if the temperature remains elevated for less than 5 minutes, preferably less than 3 minutes, no significant degradation will occur.

The superabsorbent polymers of this invention are useful in the manufacture of moisture absorbent articles, such as disposable diapers, sanitary napkins, incontinence garments and bandages. The superabsorbent compositions of this invention are particularly useful in the manufacture of thin and ultra thin disposable diapers which have excellent moisture absorbence capacity, fluid distribution properties and reduced leakage.

The absorbent articles of the invention may comprise from [illegible] percent to 95 percent by weight of the superabsorbent polymers of the invention. In a typical absorbent article the superabsorbent polymer of the [illegible] be dispersed in a fiber matrix in which the [illegible] is present in an amount from [illegible] to 70 [illegible] and the fiber matrix comprising 70 to 30 [illegible] of the article. In another form of absorbent article the superabsorbent may be present in a containment structure in which the superabsorbent polymer is present in an amount of 30 to 95 percent by weight. Combinations of dispersed superabsorbent polymer and contained superabsorbent polymer are also known.

The superabsorbent polymers of this invention can be used in the manufacture of absorbent articles such

as those described in U.S. Patents 3,669,103; 3,670,731; 4,654,039; 4,699,823; 4,430,086; 4,973,325; 4892,598; 4,798,603; 4,500,315; 4,596,567; 4,676,784; 4,938,756; 4,957,590; 4,935,022; 4,673,402; 5,061,259; 5,147,343; 5,149,335; and 5,156,902.

The process used to measure the amount of caking at a particular moisture level is called the "Caking Test." Utilization of this test allows one to determine the effectiveness of an anticaking agent by determining the amount of moisture a sample may absorb and still be free-flowing. The test involves placing a desiccator, for example, a Cole-Parmer catalog #G-08904-00, into an oven. The desiccator is maintained at 80 percent humidity by placing a straight-sided evaporation dish containing a saturated solution of potassium bromide inside the dessicator. The oven is maintained at 40°C. Five-gram samples of each dried, that is, less than 5 percent water by weight, superabsorbent polymer to be tested are placed in an aluminum foil pan or plastic weigh dish measuring 6 centimeters in diameter and 1.5 centimeters deep. The pan is removed from the dessicator after an amount of time sufficient for the samples to reach the desired moisture level to test. The samples are weighed to determine the moisturized mass and then allowed to cool to room temperature. While gently agitating, for example, by lightly tapping, the sample is screened through a No. 4 U.S. [illegible]ndard Testing Sieve ([illegible] millimeters). The [illegible] passes through the sieve is weighed to [illegible] mass of the non-caking polymer particles. [illegible] the particles which are non-caking is then [illegible] the following formula: Non-caking percent = (mass of non-caking polymer particles/moisturized mass) x 100. The moisture level in percent may be determined by the following formula [(moisturized mass - weight of initial sample)/weight of initial sample)] x 100.

The "Dust Test," that is, the process for measuring the amount of unassociated dust having a diameter less than a predetermined size present in a

water-swellable polymer composition, is described below. In the case of the examples, the measurement process was used to determine the amount of unassociated matter having a maximum size less than or equal to 10 microns. The process is applicable, however, to measure the amount of unassociated dust having a diameter less than other predetermined sizes, the predetermined sizes typically consistent with either the manufacturer's or the purchaser's specifications. The process comprises:

(a) placing the composition into the sample holder of a pulsed-jet disperser;

(b) blasting the composition with air dried by passage through dehydration means; and

(c) determining the weight of particles in each of a plurality of size increments.

Dehydration means are suitable to reduce the amount of water present in the air to be used to blast the polymer composition. Suitable dehydration means include molecular sieve cartridges, dessicating materials and membranes. An especially preferred dehydration means is a molecular sieve cartridge.

The dust levels of the coated samples may be measured as follows. Approximately 0.01 to 0.2 grams of the sample to be tested is placed in the sample holder of [illegible] disperser, for example, an AEROSIZER™ [illegible] from Amherst Process Instruments, Hadley, [illegible]). The pulsed-jet disperser is fitted with a [illegible] screen to prevent particles greater than 250 microns in diameter from entering the detector. The air delivered to the pulsed-jet disperser is filtered using the factory supplied filter and is dried using a molecular sieve cartridge. The samples are blasted with the dried air, with the exhaust stream being directed to the detector and analyzed until the instantaneous count rate decays to less than 10 particles per second on the low sensitivity setting of the detector.

Measurements are made on separate portions of the samples and are averaged. For each portion, a determination is made of the total number of particles in each of 500 size increments evenly distributed logarithmically from 0.2 to 200 microns, the instrument exhibiting an approximate lower level of detection of 0.5 microns. The weight of material in each of these size increments is calculated using the following equation:

Weight = (number of particles in an increment)(density)(π)(particle diameter)^3/6.

For sodium polyacrylate superabsorbent materials, the density is assumed to be 1.60 g/cm^3.

The aggregate weight of unassociated material less than 10 microns is designated weight$_{[illegible]}$. This weight is compared to the weight of sample originally introduced into the disperser, that is weight$_{[illegible]}$. The percent dust is determined in accordance with the following equation:

Weight percent dust (%) = [100(weight$_{[illegible]}$)/(weight$_{[illegible]}$)]. [illegible]

The "Surface Tension Test" utilizes a Du Nouy tensiometer, for example, a Processor Tensiometer K12™ from Kruss. The preparation of an aqueous fluid to be measured in equilibrium with the absorbent polymer and [illegible] has been substantially adapted from U.S. Patent [illegible] [illegible] gram extracts of each of the above [illegible] polymers, that is, the control (untreated) [illegible] polymer and the absorbent polymer treated with [illegible] reducing agents, are shaken with 200 grams of a 0.9 weight percent NaCl solution to simulate a urine solution. The surface tension is then measured with the tensiometer.

Example [illegible]

A test was conducted to examine the non-caking percent, moisture level in percent, and dustiness of

water-swellable polymers and the surface tension of a liquid in contact with said polymers.

A water-swellable polymer was obtained which had been dried to about 5 percent moisture. The polymer was DRYTECH™ 2035 superabsorbent (available from The Dow Chemical Company), a heat-treated, 62 percent neutralized polyacrylate. A predetermined amount of anticaking agent, as shown in Table I, dissolved in isopropyl alcohol except where indicated, was added dropwise to samples of the polymer at 25°C. The coated particles were then rolled for 60 minutes to distribute the agent on the particle surfaces. The non-caking percent and moisture level (in percent) during the Caking Test were measured as described in the Caking Test above. The amount of dust below 10 microns was measured as described in the Dust Test above. The surface tension was measured as described in the Surface Tension Test above. The results with respect to each sample are set forth in Table I. Table Ia describes the surfactants used as anticaking agents exemplified in Table I. ARQUAD™ and ETHOQUAD™ are trademarks of Akzo Nobel Chemicals Inc. KEMAMINE™ is a trademark of Humko Chemical Division, Witco Corporation.

TABLE I

Antica[illegible]	Amount of Anticaking Agent (ppm based on polymer)	Non-caking percent (%)	Moisture Level Percent (%)	Amount of Dust (ppm based on polymer)	Fluid Surface Tension (dynes/cm)
CO[illegible]	0.000	0.00%	24.60%	53.	66.2
ARQUAD[illegible]	1000	99.84%	22.00%	0.42	65.2
ETHOQUAD™ T/12-75 (2)	1000	99.07%	28.40%	0.68	65.1
ETHOQUAD™ O/12 (3)	1000	99.38%	30.00%	7.8	67.0
ARQUAD™ 12-33 (4)	1000	89.59%	23.00%	2.6	64.9
ARQUAD™ 16-50 (5)	1000	100.00%	24.60%	3.3	61.9
ARQUAD™ 18-50 (6)	1000	99.84%	25.00%	4.7	68.0
ARQUAD™ S 50 (7)	1000	97.22%	29.60%	0.091	65.3
ARQUAD™ S-50 (7)	1000	96.76%	29.60%	0.45	63.2
Kemamine™ Q2983 C (8)	1000	99.03%	24.20%	0.23	69.7
Kemamine™ Q2983-C (8)	300	100.00%	20.20%	9.1	66.4
ARQUAD™ 12 37W (9)	1000	100.00%	21.20%	0.50	62.4
ARQUAD™ 12-37W (9)	750	99.83%	20.20%	0.16	62.6
ARQUAD™ 12-37W (9)	500	99.83%	19.40%	0.53	63.6
ARQUAD™ T-27W (10)	1000	100.00%	21.60%	0.31	62.9
ARQUAD™ T-27W (10)	750	99.67%	20.80%	1.3	62.1

TABLE II (continued)

Anticaking Agent	Amount of Anticaking Agent (ppm based on polymer)	Non-caking percent (%)	Moisture Level Percent (%)	Amount of Dust (ppm based on polymer)	Fluid Surface Tension (dynes/cm)
ARQUAD[illegible]	500	99.67%	20.60%	0.53	63.4
5% Active ARQUAD[illegible] H_2O (11)	1000	85.99%	25.60%	0.54	68.2
ARQUAD™ [illegible] (11)	1000	99.50%	20.20%	0.89	63.3
ARQUAD™ HTL8-MS (12)	1000	100.00%	24.80%	0.094	68.1
ARQUAD™ HTL8-MS (12)	300	77.13%	26.80%	13.	66.2
ARQUAD™ HTL8-MS (12); 600 ppm Mineral Oil Added	1000	99.84%	25.40%	0.25	64.0
ARQUAD™ HTL8-MS (12), 600 ppm Mineral Oil Added	300	100.00%	21.40%	0.145	65.9
Kemamine™ Q9702-C (13)	1000	98.72%	25.20%	0.11	62.9
2% of 5% Active Q9702-C™ (13) in H_2O	1000	99.83%	20.00%	0.65	62.7
Kemamine™ Q2802-C (14)	1000	96.72%	22.00%	0.095	63.3
2% of 5% Active Q2802-C™ (14) in H_2O	1000	100.00%	20.40%	0.56	63.1

TABLE IA
DESCRIPTION OF QUATERNARY SURFACTANTS

	PRODUCT NAME	COMMON NAME OF QUATERNARY SURFACTANT
(1)	Arquad™ 2C-75	Di(cocoalkyl)dimethyl quaternary ammonium chloride
(2)	Ethoquad™ T/12-75	Tallowalkylmethyl di(2-hydroxyethyl) ammonium chloride
(3)	Ethoquad™ O/12	Oleylmethyldi(2-hydroxyethyl) ammonium chloride
(4)	Arquad™ 12-23	Dodecyltrimethyl ammonium chloride
(5)	Arquad™ 16-50	Hexadecyltrimethyl ammonium chloride
(6)	Arquad™ 18-50	Octadecyltrimethyl ammonium chloride
(7)	Arquad™ S-50	Soyaalkyltrimethyl ammonium chloride
(8)	Kemamine™ Q-2983-C	Erucyltrimethyl ammonium chloride
(9)	Arquad™ 12-37W	Dodecyltrimethyl ammonium chloride
(10)	Arquad™ T-27W	Tallowalkyltrimethyl ammonium chloride
(11)	Arquad™ 2H[illegible]PG	Di(hydrogenated tallowalkyl)dimethyl quaternary ammonium chloride
(12)	Arquad™ HTL8-MS	2-Ethylhexylhydrogenatedtallowtrimethyl ammonium methyl sulfate
(13)	Kemamine™ Q-9702-C	Di(hydrogenated tallowalkyl)dimethyl quaternary ammonium chloride
(14)	Kemamine™ Q-2802-C	Dibehenyldimethyl ammonium chloride

Example II

A test was conducted to examine the non-caking percent, moisture level in percent, and dustiness of water-swellable polymers and the surface tension of a liquid in contact with said polymers.

A water-swellable polymer was obtained which had been dried to about 5 percent moisture. The polymer was DRYTECH™ 2035 superabsorbent (available from The Dow Chemical Company), a heat-treated, 62 percent neutralized polyacrylate. The polymer was heated to 50°C in a 3-liter twin shaft, counter-rotating, batch paddle blender. Agitation was started and a predetermined amount of anticaking agent and/or dedusting agent, as shown in Table II, the anticaking agent being dissolved in the dedusting agent except where indicated, was sprayed on the polymer using an air atomizing nozzle. Agitation was continued until the mixture was non-cohesive, from 2 to 15 minutes. The non-caking percent and moisture level in percent is measured as described in the Caking Test above. The amount of dust below 10 microns was measured as described in the Dust Test above. The surface tension was measured as described in the Surface Tension Test above. The results with respect to each sample was set forth in Table II. In Table II, 09702 represents Kenamine™ [illegible], 02902 represents Kenamine™ 2902-C, Voranol represents [illegible] polyether polyol available from The Dow [illegible]pany, and "M.O." represents mineral oil.

[illegible]

TABLE 11

Anticaking Agent	Anticaki... Agent (ppm bas... on polyme...	...ng Agent	Dedusting Agent (ppm based on polymer)	Added Water (%)	Non-caking percent (%)	Moisture Level Percent (%)	Amount of Dust (ppm based on polymer)	Surface Tension (dynes/cm)
none	0	none	0	0	0.15	33.60	55.	67.7
none	0	Voranol	700	2.5	0.00	34.40	0.018	63.2
Q9702C (1)	1000	Voranol	334	2.5	100.0	33.40	0.14	65.1
Q9702C (1)	500	Voranol	167	2.5	100.00	32.40	0.53	68.6
Q9702C (1)	200	Voranol	67	2.5	97.12	32.00	1.6	68.9
Q9702C (1)	100	Voranol	33	2.5	99.85	33.40	6.8	70.1
Q9702C (1)	100	Voranol/M.O	400/33	2.5	99.51	22.40	1.3	66.8
Q9702C (1)	100	Voranol	433	2.5	99.17	21.20	0.16	68.2
none	0	none	0	0	0.59	35.80	22.	70.5
none	0	Voranol	600	3	1.04	35.00	0.12	61.7
Q2802C (2)	1000	Voranol	334	2.5	99.09	32.40	0.061	66.3
Q2802C (2)	500	Voranol	167	2.5	83.43	34.00	1.5	68.1
Q9702C (1)	100	Voranol	500	3	99.70	33.80	0.55	64.5
Q9702C (1)	150	Voranol	450	3	99.70	34.40	0.8	66.9

(1) Di(hydrogenated tallow alkyl)dimethyl quaternary ammonium chloride
(2) Dibehenyldimethyl ammonium chloride

The anticaking absorbent polymers of the examples exhibit centrifuge capacities and absorbences under load (AUL) similar to untreated absorbent polymers. In view of the description and examples, other embodiments will be readily ascertained by one having skill in the art. Accordingly, the scope of the invention shall be limited only by the claims set forth below.

CLAIMS:

1. A non-dusty, anti-caking composition comprising:

a) water-absorbent lightly crosslinked water-insoluble polymer particles;

b) an effective amount of an anticaking agent; and

c) from 100 ppm to 6000 ppm based on the weight of the polymer particles of a hydrophobic dedusting agent.

2. A non-dusty, anti-caking composition comprising:

a) water-absorbent lightly crosslinked water-insoluble polymer particles;

b) an effective amount of an anticaking agent; and

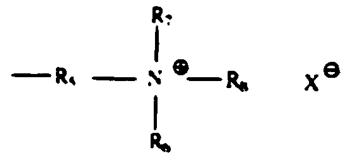
c) from 100 ppm to 3000 ppm based on the weight of the polymer particles of a hydrophilic dedusting agent.

3. The composition of Claims 1 or 2 wherein the anticaking agent is represented by the formula I

(I)

wherein R and R are independently a C-C aliphatic hydrocarbon, a C-C aliphatic hydrocarbon substituted with an unsubstituted aryl group, a C-C aliphatic hydrocarbon substituted with an aryl group substituted with a C-C aliphatic hydrocarbon, or a phenyl group; R is a C-C aliphatic hydrocarbon or a phenyl group; and R is a C-C aliphatic hydrocarbon, a C-C aliphatic hydrocarbon

substituted with an unsubstituted aryl, or a C_1-C_4 aliphatic hydrocarbon substituted with an aryl group substituted with a C_1-C_{22} aliphatic hydrocarbon, or R_4 is a radical represented by the formula II:



(II)

wherein R_5 is a C_1-C_6 alkylene group; R_6 is a C_1-C_4 aliphatic hydrocarbon or a phenyl group; R_7 and R_8 are independently a C_1-C_{22} aliphatic hydrocarbon, a C_1-C_4 aliphatic hydrocarbon substituted with an unsubstituted aryl group, a C_1-C_4 aliphatic hydrocarbon substituted with an aryl group substituted with a C_1-C_{22} aliphatic hydrocarbon, or a phenyl group; or R_7 and R_8 taken together with the nitrogen atom forms a ring selected from the group consisting of pyrrolidine, pyrroline, pyrrole, pyrazole, triazole, indole, piperazine, piperidine, morpholine and hexahydroazepine; or R_6, R_7 and R_8 taken together form a ring selected from the group consisting of pyridinium and imidazolinium, and $X^{\ominus}$ is an anion; with the proviso that if R_4 is not the radical represented by formula II then the sum of the carbons in R_1, R_2, R_3 and R_4 must be at least 15 and if R_4 is the radical represented by formula II then at least one of R_1, R_2, R_3 and R_4 must have 8 or more carbon atoms.

The composition of any of the preceding ...in the anticaking agent is dihydrogenated ...yl ammonium chloride.

5. The composition of any of the preceding claims wherein the composition comprises:

...water-absorbent lightly crosslinked water-... insoluble polymer particles.

b) from 60 ppm to 150 ppm, based on the weight of the polymer particles, of dihydrogenated tallow dimethyl ammonium chloride; and

c) from 400 ppm to 600 ppm, based on the weight of the polymer particles, of a polyether polyol.

6. An absorbent article prepared from the composition of Claims 1 or 2.

7. A process comprising:

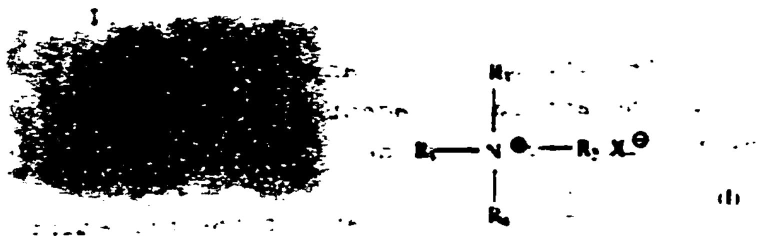
a) preparing a water-swellable hydrogel by a gel polymerization process;

b) drying and sizing the thus prepared hydrogel to obtain a composition comprising dried and sized particles; and

c) contacting the composition with an effective amount of an anticaking agent.

8. The process of Claim 7 wherein the composition is heat treated before contacting the composition with the anticaking agent.

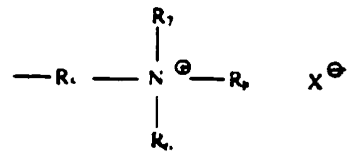
9. The process of any of the preceding claims wherein the anticaking agent is represented by the formula I



(I)

wherein R_1 and R_2 are independently a C_1-C_{22} aliphatic hydrocarbon, a C_1-C_4 aliphatic hydrocarbon substituted with an unsubstituted aryl group, a C_1-C_4 aliphatic hydrocarbon substituted with an aryl group substituted with a C_1-C_4 aliphatic hydrocarbon, or a phenyl group; R_3 is a C_1-C_4 aliphatic hydrocarbon or a phenyl group; and R_4 is a C_1-C_{22}

aliphatic hydrocarbon, a C_1-C_4 aliphatic hydrocarbon substituted with an unsubstituted aryl, or a C_1-C_4 aliphatic hydrocarbon substituted with an aryl group substituted with a C_1-C_{22} aliphatic hydrocarbon, or R_4 is a radical represented by the formula II:



(II)

wherein R_5 is a C_1-C_4 alkylene group; R_6 is a C_1-C_4 aliphatic hydrocarbon or a phenyl group; R_7 and R_8 are independently a C_1-C_{22} aliphatic hydrocarbon, a C_1-C_4 aliphatic hydrocarbon substituted with an unsubstituted aryl group, a C_1-C_4 aliphatic hydrocarbon substituted with an aryl group substituted with a C_1-C_{22} aliphatic hydrocarbon, or a phenyl group; or R_7 and R_8 taken together with the nitrogen atom forms a ring selected from the group consisting of pyrrolidine, pyrroline, pyrrole, pyrazole, triazole, indole, piperazine, piperidine, morpholine, and hexahydroazepine, or R_6, R_7, and R_8 taken together form a ring selected from the group consisting of pyridinium and imidazolinium; and $X^{\ominus}$ is an anion; with the proviso that if R_4 is not the radical represented by formula II then the sum of the carbons in R_1, R_2, R_3, and R_4 must be at least 15 and if R_4 is the radical represented by formula II then at least one of R_1, R_2, R_3, and R_4 must have 8 or [illegible] atoms.

[illegible] A non-dusty, anti-caking composition [illegible] by contacting water-absorbent lightly crosslinked water insoluble polymer particles with an effective amount of an anticaking agent and at least one of

a) from 100 ppm to 6000 ppm based on the weight of the polymer particles of a hydrophobic dedusting agent; and

b) from 100 ppm to 3000 ppm based on the weight of the polymer particles of a hydrophilic dedusting agent.

INTERNATIONAL SEARCH REPORT

International Application No
PCT/US 97/05055

A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 A61L15/60 A61L15/48 A61L15/28

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 A61L

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practical, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	EP 0 509 708 A (DOW CHEMICAL CO) 21 October 1992 see page 3, line 52 - line 54 see page 4, line 53 - page 5, line 4 ---	1,2
Y	US 4 997 714 A (FARRAR DAVID ET AL) 5 March 1991 cited in the application see column 1, line 35 - line 54 see column 2, line 7 - line 12 see column 3, line 1 - line 3 see column 6, line 46 - column 7, line 2 see claims; examples ---	1-10

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[X] [illegible] continuation of box C.

[X] Patent family members are listed in annex.

[illegible]

"A" [illegible] art which is not [illegible]

"E" earlier document but published on or after the international filing date

"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)

"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention

"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone

"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art.

"&" document member of the same patent family

Date of the actual completion of the international search	Date of mailing of the international search report
21 August 1997	27-08-1997

Name and mailing address of the ISA	Authorized officer
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International Application No
PCT/US 97/05055

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